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MISSOURI DEPARTMENT OF TRANSPORTATION MATERIALS ENGINEERING Jefferson City, Missouri

Test Method MoDOT T26 DETERMINATION OF CALCIUM CARBONATE AND MAGNESIUM CARBONATE IN LIME AND PIGMENT MATERIALS

1.0 SCOPE.

1.1 This method describes a procedure for determining the percent Calcium Carbonate and percent Magnesium Carbonate in Agricultural Lime and Calcium Carbonate paint pigments.

2.0 REAGENTS AND APPARATUS.

- 2.1 (a) Sargent Malmstadt Automatic Spectro-Electro titrator, Model S-29700
 - (b) Hexaver Solution
 Dissolve 65 gm Hexaver (Disodium Dihydrogen 1, 2,
 Cyclohexanediaminetetracetate) in 2.0 liters of H₂O
 - (c) Magnesium Chloride Solution
 Dissolve 8.00 gm MgCl₂.6H₂O Reagent Grade) in H₂O and dilute to 1 liter
 - (d) Calcon Indicator
 Dissolve 0.30 gm Calcon in 50 ml of Methanol
 - (e) EBT Indicator
 Dissolve 0.30 gm of Erichrome Black T in 50 ml of Methanol
 - (f) Potassium Hydroxide Solution
 Dissolve 100 gm KOH (Reagent Grade) in 200 ml H₂O

3.0 STANDARDIZATION OF HEXAVER SOLUTION.

3.1 (a) Weigh 0.5801 gm Calcium Carbonate (Primary Standard Grade) and transfer to a 500 ml volumetric flask. Slowly add 15 ml HCL (Sp.Gr. 1.19), and boil for a few minutes to expel CO₂. Add 2 gm NH₄CL (Reagent Grade) and 200 ml H₂O. Add with a pipette 10.00 ml of the MgCl₂ solution, and make alkaline to methyl red with NH₄0H (Sp.Gr. 0.90). Cool to room temperature and dilute to volume.



(b) Turn on the power switch of the automatic titrator and allow to warm up for about 15 minutes. Set the controls as follows:

Function Switch - Spectro Polarity Switch - No. 2 Wavelength Selector - 650 The Hupp Cadmium Sulfide photocell should be used.

Pipette 25.00 ml aliquots into two 100 ml tall form beakers. To one beaker add 3 ml KOH Solution, 10 ml $\rm H_2O$, and 10 drops of Calcon indicator. Place the beaker on the titration platform and start the titrator. The burette should be adjusted so that the rate of delivery is about 45 seconds between the 35 ml mark and the 45 ml mark. When the titrator shuts off, record the burette reading as Tca. To the second beaker, add 10 ml NH4OH and 8 drops of EBT Indicator. Titrate as described above, and record the burette reading as Tmg.

Calculate the Calcium and Magnesium equivalents of the Hexaver as follows:

CaO Equiv.
$$(F_{ca})$$
 = $\underline{65}$ T_{ca}

MgO Equiv. (Fmg) = \underline{F}_{ca} 1.391

K = T_{mg} - T_{ca}

4.0 PROCEDURE.

4.1 Weigh 0.5000 gm sample of the material and transfer to a 250 ml beaker. Moisten with H₂O and add 10 ml HCL. Remove the insoluble matter, SiO₂, and R₂O₃ by the methods set forth in ASTM C25. Collect all the filtrates and washings from the R₂O₃ filtration in a 500 ml volumetric flask. Pipette 10 ml MgCl₂ solution into the flask, cool and dilute to volume.

Tritrate 25 ml aliquots for calcium and magnesium as described above in Section 3. Record the burette readings as $T_{\rm ca}$ and $T_{\rm mg}$

5.0 CALCULATIONS.

5.1 Calculate the percent Calcium Carbonate and percent Magnesium Carbonate as follows:

$$\% \ \, \text{CaCO}_3 \qquad = \quad F_{\text{ca}} \ \, x \ \, T_{\text{ca}} \, x \ \, 1.7848 \\ \% \ \, \text{MgCO}_3 \qquad = \quad F_{\text{mg}} \, x \, (T_{\text{mg}} \, \text{--} \, T_{\text{ca}} \, \text{--} \, K) \, x \, 2.0915$$

Report the results, to the nearest 0.1 percent, as follows: % Calcium Carbonate CaCO₃) % Magnesium Carbonate (MgCO₃)

